7-Amino-4,6-Dinitrobenzofuroxan, an Insensitive High Explosive

by W. P. Norris Research Department

JUNE 1984

NAVAL WEAPONS CENTER CHINA LAKE, CALIFORNIA 93555



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FOREWORD

The performance of warheads in ordnance systems is maximized in terms of many variables including the properties of the explosive charge. Research on energetic materials at NWC seeks new explosives with superior properties for use in warheads. 7-Amino-4,6-dinitrobenzofuroxan is an easily prepared insensitive high explosive with calculated explosive power superior to TNT and equal to that of TATS. It is a valuable addition to the list of energetic materials which may be useful for warhead design.

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Approved by E. B. ROYCE, Head Research Department 30 March 1984 Under authority of
K. A. DICKERSON
Capt., U.S. Navy
Commander

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(U) 7-Amino-4,6-dinitrobenzofuroxan is an insensitive explosive (equal to TNT in impact sensitivity) with a calculated detonation velocity equal to that of TATB. A simple synthesis route is described. The position of the amino group is proved by reduction with triphenyl-phosphine to give 7-amino-4,6-dinitrobenzofurazan, a known compound. A by-product isolated from the induction reaction is 2-(triphenylphosphinimido)-4,6-dinitroaniline.

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INTRODUCTION

This report describes the explosive and physical properties, synthesis, and structure proof of 7-amino-4,6-dinitrobenzofuroxan (la).

EXPLOSIVE AND PHYSICAL PROPERTIES

7-Amino-4,6-dinitrobenzofuroxan is an insensitive, thermally stable explosive. It is a fairly dense, easily prepared compound with a calculated detonation velocity equivalent to that of 1,3,5-triamino-2,4,6-trinitrobenzene (TATB) (see Table 1).

The introduction of an amino group into 4,6-dinitrobenzofuroxan (DNBF) has a remarkable effect upon physical and explosive properties. The melting point, density, and calculated detonation velocity all increase significantly and the impact sensitivity is reduced dramatically, as shown in Table 2.

AT A SAN DESCRIPTION OF SAN DESC

TABLE 1. Properties of 7-Amino-4,6-dinitrobenzofuroxan.

Properties	Measurements
Molecular formula	C ₆ H ₃ N ₅ O ₆
Molecular weight	241.12
Density ^a	1.902 ±0.008 g/cm ³
Melting point (DSC, 10°/min)b	270° (decomposition)
Oxygen balance (CO)	-10
Percent nitrogen	29.1
Detonation velocity (calculated) ^C	7.91 mm/us
Detonation pressure (calculated)c	282 Kbar
Impact sensitivity (H ₅₀)d	53 cm (TNT = 54 cm)
Heat of formation	+36.79 ±0.72 Kcal/mo

^a Gas comparison pycnometer, type 6102-12, System Science and Software, LaJolla, Calif.

TABLE 2. Comparison of Properties of DNBF and la.

Properties	DNBF	la
Melting point Density	174-175° 1.747 ±0.001 g/cm ³	270° (decomposition) 1.902 ±0.008 g/cm ³
Impact sensitivity Detonation velocity (calc.)	18 cm 7.71 mm/μs	53 cm 7.91 mm/μs

SYNTHESIS

A synthesis of a monoamino-4,6-dinitrobenzofuroxan (1), by the thermal decomposition of 1-azido-3-amino-2,4,6-trinitrobenzene, was reported in 1968 (Reference 2). The compound was fully characterized, but the position of the amino group in the compound was not established.

b See Figure 1.

^c See Reference 1.

d Bureau of Mines design instrument, type 12 tools, 2.5 Kg wt.

A synthetic approach which would seemingly establish the correct structure of 1 would be the treatment of 7- or 5-chloro-4,6-dinitrobenzofuroxans (2 or 3) (Reference 3) with ammonia to give the 7- or

$$\begin{array}{c|c}
 & NO_2 \\
 & NH_3 \\
\hline
CH_2Cl_2 \\
\hline
 & NH_2 \\
\hline
 & NH_2 \\
\hline
 & (100X yield)
\end{array}$$

5-amino-4,6-dinitrobenzofuroxans (la or lb). However, treatment of either 2 or 3 with ammonia gives the same product.

STRUCTURE PROOF

Molecular rearrangement may occur in compounds with a nitro group adjacent to a furoxan ring which interconverts the 5 and 7 positions (Reference 4). Thus, an approach other than simple substitution is

required to establish the position of the amino group in 1. Reduction of the furoxan ring to a furazan ring prevents this kind of isomerization. Treatment of 1 with triphenylphosphine, a reducing agent that converts 4,6-dinitrobenzofuroxan to the corresponding furazan derivative (Reference 5), gives 4. The reduction product, 4, was also synthesized

$$O_{2}NH_{2} \longrightarrow O_{2}N \longrightarrow O_{2}$$

by an independent route from the known 7-methoxy-4,6-dinitrobenzofurazan (Reference 6), 6, so its structure is definitely established as 7-amino-4,6-dinitrobenzofurazan.

7-Methoxy-4,6-dinitrobenzofurazan, 6, is prepared by acidification of the Meisenheimer complex, potassium 7,7-dimethoxy-4,7-dihydro-4,6-dinitrobenzofurazanide, 7, the structure of which has been determined by X-ray crystallography (Reference 7).

An uncertainty remaining concerns a possible retro-Boulton-Katritzky rearrangement (Reference 8)* of la at 140°C, the temperature of the reduction reaction, which conceivably could give the isomeric reduction product. However, a thermal analysis of la shows a perfectly straight heat flow versus temperature trace from 25 to 240°C which means, at least in the solid phase, there is no retro-Boulton-Katritzky rearrangement occurring below 240°C and certainly not at 140°C. The aforementioned uncertainty is thus virtually eliminated.

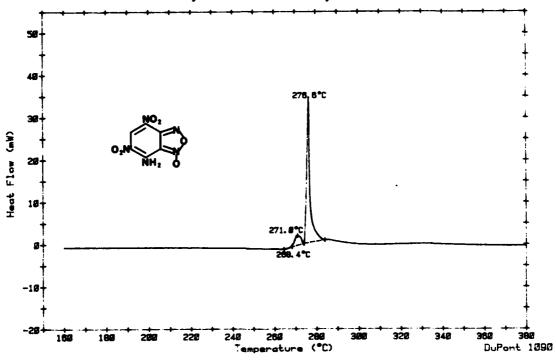


FIGURE 1. Thermal Analysis of la.

^{*}Ghosh showed that 7-anilino-4-nitrobenzofuroxan changes to 5-anilino-4-nitrobenzofuroxan above 150° in the solid state. This is the retro-Boulton-Katritzky rearrangement. The 5-anilino isomer in solution at 25° undergoes the normal Boulton-Katritzky rearrangement to 7-anilino isomer.

The thermal analysis trace above 240°C, see Figure 1, possibly represents a retro-Boulton-Katritzky rearrangement process which, above 268°C, becomes a combination rearrangement-decomposition process. At 268°C, thermal contribution from exothermic decomposition exceeds endothermic rearrangement contribution causing the line to rise until at 271°C the endothermic process again prevails and the line drops. At about 274°C the decomposition process becomes predominant. The decomposition process may involve largely the rearranged isomer, 5-amino-4,6-dinitrobenzofuroxan.

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There is another reduction product isolated. To this compound the structure, 2-(triphenylphosphinimido)-4,6-dinitroaniline, 5, is assigned. This structure is consistent with the elemental analysis, molecular weight, $^1\mathrm{H}$ and $^{13}\mathrm{C}$ NMR spectra, and the infrared spectrum of 5. The infrared absorption spectrum of 5 shows peaks at 3330 and 3210 cm $^{-1}$ for the NH₂ group and after exchange with D₂O shows peaks at 2550 and 2400 cm $^{-1}$ for the ND₂ group. Treatment of a chloroform solution of 5 (red in color) with gaseous hydrogen chloride forms gold colored plates, presumably the hydrochloride. Coupling of the two meta aromatic protons of 5 in the $^1\mathrm{H}$ NMR spectrum gives doublets for H-5 and H-3, J_{3,5} = 2.4 Hz, and the H-3 doublet is split again by phosphorous in the adjacent triphenylphosphinimido group, J_H, p = 1.1 Hz. The $^{13}\mathrm{C}$ NMR spectrum shows a C-2 doublet, J_C, p = 2.3 Hz, for $^{13}\mathrm{C}$ coupling with phosphorous through nitrogen.

CONCLUSION

A new, easily synthesized, insensitive, high explosive, 7-amino-4,6-dinitrobenzofuroxan, is described. Its structure is proved by its reduction with triphenylphosphine to 7-amino-4,6-dintirobenzofurazan, a compound of proven structure. Another reduction product, 2-(triphenyl-phosphinimido)-4,6-dinitroaniline is isolated and identified.

EXPERIMENTAL

PREPARATION OF 7-AMINO-4,6-DINITROBENZOFUROXAN (1a)

A solution of 5.00 g (0.0192 mol) of 7-chloro-4,6-dinitrobenzo-furoxan³ in 150 ml of $\mathrm{CH_2Cl_2}$ at 25°C is stirred under an ammonia atmosphere for 30 min. An orange colored solid begins separating immediately. At the end of the ammonia treatment, the orange solid is filtered off. Stirring the product in 100 ml of 3N HCl for 30 min and filtering gives 4.66 g (100% yield) of la, with a melting point of 270°C (decomposition) (see Figure 1). Recrystallization from $\mathrm{CH_3CN}$ gives 3.20 g of thin gold colored plates. Recrystallization from 70% nitric acid gives orange colored tabular crystals.

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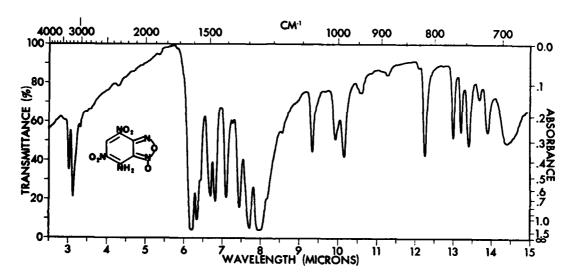
Analysis calculated for $C_6H_3N_5O_6$: C, 29.89; H, 1.25; N, 29.05. Found: C, 29.87; H, 1.28; N, 28.99.

Infrared spectrum: See Figure 2.

Mass spectrum: See Figure 3.

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¹H NMR (DMSO-d₆ + trace HCl, 30°) δ 10.10, 9.45 (NH₂, s, nonequivalent); 9.00 (H-5,s). ¹³C NMR (DMSO-d₆, TMS standard) δ 120.4 (C-4, s); 132.5 (C-5, s); 121.6 (C-6, s); 142.8 (C-7, s); 110.9 (C-8, s); 146.2 (C-9, s).



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FIGURE 2. Infrared Spectrum of 7-Amino-4,6-dinitrobenzofuroxan (la).

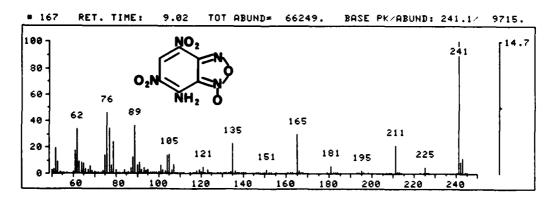


FIGURE 3. Mass Spectrum of 7-Amino-4,6-dinitrobenzofuroxan (1a).

REACTION OF 5-CHLORO-4,6-DINITROBENZOFUROXAN (3) WITH AMMONIA

A solution of 0.562 g (0.00216 mol) of 5-chloro-4,6-dinitrobenzo-furoxan in 25 ml of CH_2Cl_2 cooled to -10°C, is treated with gaseous NH $_3$, with stirring. An orange colored solid separates immediately. After 10 min, the suspended solid appears orange-red in color and the liquid phase is likewise orange-red in color. The solid product is filtered off and stirred in 50 ml of 3N HCl at 25°C to give 0.237 g (46% yield) of a tan powder with an infrared spectrum identical to that of la.

PREPARATION OF 7-AMINO-4,6-DINITROBENZOFURAZAN (4)

A solution of 0.595 g (0.00232 mol) of 7-methoxy-4,6-dinitrobenzo-furazan (Reference 6) in 10 ml of $\mathrm{CH_2Cl_2}$ at 25°C is stirred under an ammonia atmosphere for 10 min. An orange-yellow colored solid begins separating immediately. Volatiles are removed on a rotary evaporator to leave 0.565 g of the ammonium salt of 4. This is stirred for 10 min in 50 ml of 1N HCl, filtered, washed and dried to give 0.485 g (93% yield) of 4. Recrystallization from $\mathrm{CH_3CN}$ gives 0.353 g of 4, with a melting point of 249-253°C (decomposition) [Lit. m.p. 215-245°C (decomposition) (Reference 2)].

Analysis calculated for $C_6H_3N_5O_5$: C, 32.01; H, 1.34; N, 31.11. Found: C, 32.02; H, 1.39; N, 31.04.

Infrared spectrum: See Figure 4.

 1 H NMR (DMSO-d₆ + trace HCl, 40°) & 10.91, 10.08 (NH₂, s, nonequivalent); 9.08 (H-5, s).

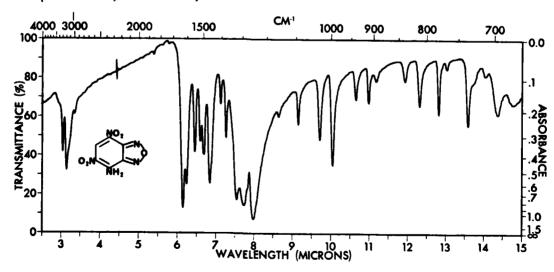


FIGURE 4. Infrared Spectrum of 7-Amino-4,6-dinitrobenzofurazan (4).

chromatography on a 35 x 210 mm silica gel column using $\mathrm{CH_2Cl_2}$ as the developing solvent. Following unreacted triphenylphosphine there are three major bands.

The first band collected gives 0.20 g of a red solid, 5. Recrystallization from CCl_4 gives red needles of 2-(triphenylphosphinimido)-4,6-dinitroaniline (5) with a m.p. of $235-236^{\circ}$.

Analysis calculated for $C_{24}H_{19}N_{4}O_{4}P$: C, 62.88; H, 4.18; N, 12.22; P, 6.76. Found: C, 62.61; H, 4.25; N, 12.05; P, 7.04.

Infrared spectrum, 3330 and 3210 cm⁻¹ (-NH₂); 2550 and 2400 cm⁻¹ (-ND₂). Mass spectrum parent ion m/e = 458. 1 H NMR (CDCl₃, 30°) $_{\delta}$ 8.48 (H-5, d, J_{3,5} 2.4 Hz); 7.8-7.5 (C₆H₅ m); 7.04 (H-3, dd, J_{3,5} 2.4 Hz, J_H, p 1.1 Hz). 13 C NMR (CDCl₃, 30°), (nitroaromatic ring) $_{\delta}$ 146.8 (C-4); 146.4 (C-6); 140.9 (C-1); 127.4 (C-5); 112.2 (C-2, d, J_C, p 10.0 Hz); 112.0 (C-3); [(C₆H₅)₃P-] $_{\delta}$ 136.3 (C-P); 132.8 (para-C, d, J_C, p 2.3 Hz); 132.5 (ortho-C, d, J_C, p 10 Hz); 129.3 (meta-C, d, J_C, p 12.3 Hz).

The second band collected gives 0.083 g of 7-amino-4,6-dinitrobenzofurazan (4). The infrared spectrum of this material is identical with the infrared spectrum, Figure 4, of the separately synthesized 7-amino-4,6-dinitrobenzofurazan (4).

The third band to be collected gives 0.10 g of triphenylphosphine oxide, with a m.p. of 156-157°. The infrared spectrum is identical to that of an authentic sample of triphenylphosphine oxide from Aldrich Chemical Co.

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     Directorate of Chemical Sciences, D. Ball (1)
1 Air Force Rocket Propulsion Laboratory, Edwards Air Force Base (AFRPL/LKLR, S. Shackelford)
1 Air Force Rocket Propulsion Laboratory, Edwards Air Force Base (AFRPL/MKL/MS 24, R. Geisler)
1 Air Force Rocket Propulsion Laboratory, Edwards Air Force Base (AFRPL/MKPA, F. Roberts)
12 Defense Technical Information Center
1 Aerojet Strategic Propulsion Company, Sacramento, CA, Via AFPRO (R. L. Lou)
I Anal-Syn Laboratory, Inc., Paoli, PA (V. J. Keenan)
1 Atlantic Research Corporation, Alexandria, VA (M. K. King)
1 Atlantic Research Corporation, Gainesville, VA (W. D. Stephens)
1 Ballistic Missile Defense Advanced Technology Center, Huntsville, AL (D. C. Sayles)
1 Cornell University, Ithaca, NY (School of Chemical Engineering, F. Rodriguez)
1 Fluorochem, Inc., Azusa, CA (K. Baum)
1 Hercules, Incorporated, Allegany Ballistics Laboratory, Cumberland, MD (R. C. Musso)
1 Hercules, Incorporated, Eglin Air Force Base, FL, Via AFPRO (AFATL/DLDL, R. L. Simmons)
2 Hercules, Incorporated, Magna, UT
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 2 Johns Hopkins University, Applied Physics Laboratory, Laurel, MD
     Department of Chemistry, J. J. Kaufman (1)
     T. M. Gilliland (1)
 1 Lockheed Missiles & Space Company, Sunnyvale, CA (83-10, Linsk)
 4 Los Alamos National Laboratory, Los Alamos, NM
     NSP/DOD, MS 245
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    E. S. Sutton (1)
    C. W. Vriesen (1)
1 Morton-Thiokol Corporation, Government Systems Division, Ogden, UT (Technical
  Director, T. F. Davidson)
2 Morton-Thiokol Corporation, Huntsville Division, Huntsville, AL
    D. A. Flanigan (1)
    G. F. Mangum (1)
2 Morton-Thiokol Corporation, Wasatch Division, Brigham City, UT, Via AFPRO
    MS 240, G. Thompson (1)
    J. Hinshaw (1)
1 North Texas State University, Denton, TX (Department of Chemistry, A. P. Marchand)
1 Polysciences, Inc., Warrington, PA (B. D. Halpern)
2 Rockwell International Corporation, Canoga Park, CA
     Rocketdyne Division
       K. O. Christe (1)
       M. B. Frankel (1)
1 Rohm and Haas Company, Huntsville Defense Contract Office, Huntsville, AL (H. Shuey)
2 SRI International, Menio Park, CA
     C. D. Bedford (1)
     D. L. Ross (1)
1 United Technologies Corporation, Chemical Systems Division, Sunnyvale, CA (C. M. Frey)
2 University of California, Lawrence Livermore National Laboratory, Livermore, CA
     L-324, R. McGuire (1)
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